PRELIMINARY PERFORMANCE COMPARISON BETWEEN THE GLOVE BOX AND ELUTRIATOR METHODS BASED ON ANALYSES OF DUPLICATE SPLITS FROM THE NORTH RIDGE ESTATES SITE, KLAMATH FALLS, OREGON

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During their investigation of the North Ridge Estates Site in Klamath Falls, Oregon, the United States Environmental Protection Agency (EPA) prepared duplicate splits of 12 soil composites and had one sample of each pair analyzed, respectively, using the Glove Box Method (U.S.EPA 2004) and the Modified Elutriator Method (Berman and Kolk 2000).

Although 12 samples is a small data set from which to evaluate the relative performance of two methods in detail, it is large enough to provide a good, preliminary indication of any trends in such relative performance. Therefore, the results from the set of paired analyses derived from North Ridge Estates are evaluated below to compare the performance of the Glove Box Method and the Modified Elutriator Method.

Background

In response to a need to support risk-based decision making at asbestos sites, both the Glove Box Method and the Modified Elutriator Method were developed to determine asbestos concentrations in soils and bulk materials in a manner that would indicate the degree to which such materials might release asbestos into the air when disturbed by human activities or natural forces. Each method is briefly described below along with a summary of the status of knowledge concerning method performance.

The Modified Elutriator Method

The Modified Elutriator Method (Berman and Kolk 2000) is a refinement of the earlier Superfund Method (Berman and Kolk 1997). This method is designed to provide objective, quantitative determination of asbestos concentrations in soils and other bulk materials that can be combined with published dust emission and dispersion models to predict airborne asbestos exposure concentrations with reasonable accuracy (see below).

Using the method, a source material of interest is representatively sampled and the kg-sized samples are sieved through a screen with 1 cm (3/8ths inch) openings to separate a coarse and fine fraction. If necessary, the fine fraction may be further homogenized and split to obtain nominal 100-g samples that are sent to the laboratory for analysis. No other processing of samples is required.

In the laboratory, the samples are stored in a humidity controlled chamber to bring them into equilibrium with air at 50% relative humidity.

A known mass of the conditioned samples are then placed in the tumbler of a dust generator/elutriator and the samples are gently tumbled so that entrainable material is picked up by an air stream flowing through the system. The air stream passes from the tumbler to a vertical elutriator where (due to flow conditions) only respirable-sized particles pass to the top. Larger particles fall to the bottom of the elutriator.

The respirable fraction from the sample is then captured on a filter and the filter is weighed (to determine the mass of respirable dust captured on the filter) and then prepared by a direct-transfer procedure for analysis by transmission electron microscopy (TEM) to determine the concentration of asbestos captured on the filter. Results are reported as the ratio of the number of asbestos structures to the mass of respirable dust captured on the filter (str/g_{PM10}).

Based on available information, the performance of the Modified Elutriator Method can be summarized as follows:

1. ***precision***. An extensive database of paired results from duplicate splits indicate that the relative percent differences (RPD's) observed for half of such splits are less than 50% and 4/5ths of such RPD's are less than 100% (Berman 2000);

2. ***interpretation.*** By normalizing asbestos counts to dust mass (to a good approximation), measurements derived using this method represent intrinsic properties of the sampled material, just as spectroscopic measurements to determine chemical concentrations are intrinsic properties of the sampled material (for details, see Berman and Kolk 2000 and Berman 2000). This is *not* typical of measurements derived using other bulk asbestos methods. Moreover, the dimensions (units) in which measurements derived using this method are reported match those required as inputs for published dust emission models (to convert them to asbestos emission models). Thus, measurements can be combined with such models to predict exposure under any scenario for which an appropriate emission model exists; and

3. ***accuracy.*** Results of a published study comparing predicted and measured airborne exposure concentrations (Berman 2000) indicate that exposure predictions based on measurements derived using the Modified Elutriator Method (Berman and Kolk 2000) are reasonably accurate and calibration is not required.

The Glove Box Method

Although there is currently no formal documentation for the Glove Box Method, the following description is based on the information provided in a Quality Assurance Project Plan (QAPP) developed by EPA (U.S.EPA 2004) for the North Ridge Estates Site.

Using this method, samples from a source material of interest are first sieved by passing the sample through a screen with 4 mm (approximately 1/8th inch) openings. A small quantity (mass not precisely defined) of sample is then placed in a stainless steel pan inside a glove box and the material is agitated by stirring with a stainless steel spoon until visible dust is generated within the glove box. Once sufficient (criteria not specified) visible dust is generated, agitation is discontinued and, after some time is allowed for settling of larger particles (specific interval not specified), air pumps are turned on and the airborne dust within the glove box is sampled by passing air (at a known flow rate for a fixed period of time) through a filter.

Filters from the glove box are prepared by a direct transfer technique for analysis by TEM to determine the structure number concentration of asbestos in the air collected from the glove box (str/cm^3).

Based on available information, the performance of the Glove Box Method can be summarized as follows:

1. ***precision.*** Due to an apparent lack of paired measurements on duplicate splits, the precision of this method is not currently defined;

2. ***interpretation.*** As stated, the method is designed to provide a *qualitative* indication of the potential for source material to release asbestos into the air when disturbed. Neither have studies been reported nor theory been developed to link measurements derived using the glove box to exposures associated with disturbance of asbestos-containing source materials in the real world; and

3. ***accuracy.*** There are no known studies indicating the accuracy of this method.

Evaluation of Paired Analyses

Both asbestos structure counts observed using each method and the corresponding concentrations determined using each method are compared below.

Comparison by Structure Count

Results from the paired elutriator/glove box analyses conducted during the study of the North Ridge Estates Site are summarized in Table 1. In Table 1, the first two columns indicate the Sample Identification Nos. used for the elutriator analyses and the glove box analyses, respectively. Note that, although different identification numbers were applied for each set of analyses (apparently for contractual reasons), analyses reported in corresponding rows of the table represent analyses of paired, duplicate splits of sampled material.

The third column of Table 1 indicates the mass fraction of asbestos-containing material (ACM) debris observed (and removed) from each sample prior to analysis by either method. Note that the results in the table are arranged in descending order from highest to lowest ACM content.

The fourth column of the table indicates the type of asbestos observed in each sample.

The next five columns of the table present the results of structure counting derived using the Glove Box Method. These columns indicate, respectively, the analytical sensitivity achieved for the indicated analysis and the number of structures observed in each sample for a set of four different size categories: total ISO structures, total protocol structures, long protocol structures, and PCME fibers. Definitions for these size categories are provided in earlier reports from the site (see Berman 2004 and Berman 2005).

Note, although the various size categories reproduced in Table 1 were used in different ways during evaluation of exposure and risk at the site, their significance for risk assessment is irrelevant to the performance comparison being considered in this report. Therefore, the implications of the various size categories are not further addressed in this document, except to the extent that comparison across structure counts (or corresponding concentrations) for these various size categories are informative regarding the method comparison being addressed. These other size categories are included in Table 1, however, simply to facilitate a visual check that counts across size categories for any particular sample are not inconsistent when analyzed by each of the two methods.

Columns 10 through 14 of Table 1 present the results of structure counting derived using the Elutriator Method. These columns indicate, respectively, the analytical sensitivity achieved for the indicated analysis and the number of structures observed in the set of four different size categories: total ISO structures, total protocol structures, long protocol structures, and PCME fibers. Thus, the information presented in these five columns corresponds to the information reported for Glove Box analyses in the previous five columns.

To compare measurements derived, respectively, using the Glove Box Method and the Elutriator Method, the 15th column of Table 1 indicates the Relative Percent Difference (RPD) estimated for counts of total ISO structures reported for each pair of measurements indicated in each row of the table[1]. The RPD between two measurements is estimated as 100 times the absolute value of the difference between the two measurements divided by the mean of the two measurements. Thus:

$$RPD = 100*abs(a-b)/[0.5*(a+b)] \qquad \text{(Eq. 1)}$$

where:

RPD	is the Relative Percent Difference (%);
a	is the value derived for the first measurement (structure counts, in this case);
b	is the value derived for the second measurement (structure counts, in this case); and
abs	is the absolute value of the quantity indicated.

Note that, when one of two measurements being compared is zero, the calculated RPD will always be 200%, which is the maximum possible value. This is due to the manner in which RPD's are calculated.

The RPD is a measure of precision and the smaller the value of an RPD, the greater the agreement (or precision) across measurements. The RPD estimated for a pair of identical measurements (constituting perfect agreement) is zero.

As can be seen, RPD's estimated for paired Glove Box/Elutriator Method measurements presented in Column 15 of Table 1 vary between 0% and 183%. There are also four values that are indicated to be "NA" (not applicable) because one of the pair of these measurements is zero. To evaluate the degree of agreement across the two methods, the RPD's in Table 1 are compared to the range of RPD's that are typically achievable when duplicate splits are analyzed by the Elutriator Method alone (see background section above).

Based on the range of achievable RPD's reported for duplicate splits of samples analyzed using the Modified Elutriator Method (Berman 2000), if an RPD reported in Table 1 (for between-method analyses) is less than 75, it can be concluded that this indicates good agreement. If an RPD is greater than or equal to 75 but less then 150, agreement is considered to be fair. If an RPD is equal to or greater than 150, agreement across the Glove Box Method and the Elutriator Method is assumed to be poor. Such assessments of the degree of agreement are presented in the 16th column of Table 1.

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1 RPD's presented in Table 1 are calculated from counts of total ISO structures because these are the most general and, hence, the most numerous structures among the size categories observed. The greater the number of counts available for the determination of each RPD, the more robust the analysis.

The last column of Table 1 indicates by which method (Glove Box or Elutriator) a greater number of structures are observed. This is taken as an indication of the relative sensitivity achieved by each method for each of the paired analyses reported. Importantly, due to the unavoidable statistical variation associated with structure counting, small differences between counts should not be considered to be meaningful. Therefore, when agreement between the two methods is found to be good (based on the RPD), it is further concluded that the relative number of counts are not statistically distinguishable so that the two methods are assumed to exhibit approximately equal sensitivity. However, when agreement across the paired measurements is only fair (or worse), then the method that results in the greater number of structure counts is considered to be more sensitive for that pair of analyses.

As can be seen in Table 1, of the 14 comparisons reported[2], six show good agreement with approximately equal sensitivity exhibited by both methods. Of the remaining eight comparisons, two indicate greater sensitivity for the Elutriator Method and six indicate greater sensitivity for the Glove Box Method.

Among the latter comparisons, interestingly, are the two comparisons (for each of two asbestos types) among the paired sample set (Nos. 3090514-4080111). For this pair of analyses, results reported by the Glove Box Method indicate detection only of amosite asbestos. In contrast, results reported by the Elutriator Method indicate detection only of chrysotile. Thus, these results are inconsistent and the inconsistency cannot be explained by differences in sensitivity. This is because each method shows greater sensitivity for one of the two types of asbestos observed in this set of sample splits.

The data presented in Table 1 suggest generally fair agreement across analytical results using the two methods with generally similar sensitivity achieved by each, although there is some suggestion that the glove box may sometimes provide somewhat greater sensitivity, at least as each method was applied in this study. At the same time, given that a third of the results show poor agreement and that the paired analyses of at least one sample split are entirely inconsistent, further evaluation is warranted.

Comparison by Concentration

The concentrations of total ISO structures determined by analysis using the Glove Box and Elutriator Methods in this study are presented in Table 2. In Table 2, the first two columns indicate the Sample Identification Nos. used for the Elutriator analyses and the Glove Box analyses, respectively. The third column indicates the type of asbestos observed. Note that concentrations of different asbestos types are separately evaluated.

[2] Note, although only 12 paired samples were analyzed, two of the samples exhibit each of two kinds of asbestos and counts of different types of asbestos are separately compared.

The next three columns of Table 2 present results derived using the Glove Box Method. Thus, Columns 4 to 6 respectively indicate the analytical sensitivity achieved by the Glove Box for each analysis, the number of total ISO structures observed, and the corresponding concentration determined.

Note that, because the definition of analytical sensitivity is the concentration equivalent to the detection of a single structure, the total concentration in a sample is determined simply as the product of the analytical sensitivity achieved and the number of structures observed in that sample.

Columns 7 to 9 of Table 2 present corresponding results derived using the Elutriator Method. Thus these columns respectively indicate the analytical sensitivity achieved using the Elutriator Method, the number of total structures observed during analysis, and the corresponding concentration of total structures.

Importantly, concentrations determined by each of these methods cannot be directly compared. This is because the dimensions (units) of the concentrations do not match. However, trends in the relative concentrations can be evaluated. Thus, the ratios of the concentrations determined respectively by the Glove Box Method and the Elutriator Method are presented in the last column of Table 2.

Given that both the Elutriator Method and the Glove Box Method are intended to provide indications of the degree with which asbestos can be released to the air from a sampled material (when such material is disturbed), measurements derived using each method should remain approximately proportional from one sample to the next. This also means that the ratios of the paired measurements should remain approximately constant. However, the ratios of concentrations presented in the last column of Table 2 suggest otherwise. The values presented in this column range over a factor of approximately 900 (almost three orders of magnitude).

A more formal evaluation of the correlation between Glove Box and Elutriator measurements is provided in Figure 1. Figure 1 presents a plot of the concentrations determined by the Glove Box Method (on the Y-axis) against the concentrations determined by the Elutriator Method (on the X-axis). As is apparent in the figure, there is substantial scatter in the plot of these paired measurements and the slope of the best-fit trend line is very shallow.

The correlation coefficient (R^2) estimated for the paired data presented in Figure 1 is 0.0243, which is very small. The corresponding Pearson Product Moment Correlation Coefficient (the square route of R^2) is 0.156 and this can be used in a formal hypothesis test to determine whether these sample sets are independent (Lowry 2002). Given a sample size of 14 and $r = 0.156$, the P value is 0.297. Thus, the null hypothesis that these two data sets are independent cannot in any way be rejected. Clearly, there is little evidence for any correlation between these two sets of measurements.

Discussion

As previously indicated, measurements derived using the Elutriator Method exhibit good precision and have been shown to provide a quantitative indication of the potential for a sampled material to release asbestos when disturbed (see the Background Section above). Thus, because measurements derived using the Glove Box Method on paired samples have been shown to be independent of elutriator measurements (i.e. no evidence of correlation), results from this evaluation suggest that Glove Box data cannot be used to assess releasability, at least not quantitatively. This is likely due to a combination of the subjective manner in which dust is generated in the Glove Box Method coupled with the lack of control of various factors that also affect overall releasability.

The analytical sensitivity of the Glove Box Method is independent of the vigor with which the dust is generated in the box. Whatever the vigor with which the analyst stirs the source material, the analytical sensitivity is completely determined by the amount of air that is passed through the sample filter and the number of grid openings counted. Thus, (at a minimum) unless both the manner in which the sample is prepared and the manner in which the sample is stirred are rigidly standardized[3], one should not expect that comparable measurements necessarily indicate comparable potential for sampled material to release asbestos into the atmosphere in the field.

In fact, even if the two conditions described above are met, the glove box may still fail to provide a quantitative measure of the potential for sampled material to release asbestos. This is because other factors that affect releasability are not adequately controlled during glove box analysis either. These include the humidity under which the analysis is performed, the mass of sample in the pan, the size of the pan, the velocity and turbulence of air flow in the immediate vicinity of the pan, and the interval of time between dust generation and sampling. Such factors may all contribute to the variability that apparently precludes reasonable correlation between Glove Box Method measurements and measurements that are reasonable predictors of field behavior.

In contrast, the manner in which dust is generated in the Elutriator Method is objective and all of the other relevant factors described above (which affect releasability) are also controlled. Moreover, the vigor with which dust is generated is explicitly addressed in the determination of the analytical sensitivity for this method because analytical results are reported as the ratio of the number of asbestos structures to the mass of respirable dust that is simultaneously generated. Thus, in this case, the mass of respirable dust serves as a normalizing factor quantifying the vigor with which dust is generated. Under such

[3] Currently, the manner in which a sample is stirred is subjective.

circumstances, it has been demonstrated (Berman 2000) that the asbestos concentrations observed using the Elutriator Method quantitatively predict the potential with which sampled material will release asbestos into the atmosphere in the field. All that is required to predict the actual rate of release in the field is to account for the conditions under which such a release occurs. This is typically described by the emission models for which Elutriator Method measurements serve as inputs.

Conclusions

Although based on a relatively small number of samples, the evaluation presented above suggests that the current form of the Glove Box Method being developed by EPA may not be capable of providing measurements that are a reliable indicator of the relative potential for sampled material to release asbestos during disturbance under actual field conditions. The additional lack of theoretical work establishing a quantitative link between Glove Box measurements and asbestos release in the field also raises questions concerning the reliability of the method.

Given the above, if EPA is interested in continuing development of this method, it would be prudent to conduct additional studies to better characterize the performance of the Glove Box Method before attempting to incorporate measurements using the method into a risk-based decision framework that can be applied at sites where asbestos is a contaminant of concern.

Additional studies should also be conducted to evaluate whether, at a minimum, the Glove Box Method might serve as a reliable screening tool. In this latter case, it would be necessary to show that the method can reliably detect asbestos over the full range of source materials that can potentially release asbestos at unacceptable rates when disturbed in the field. As a screening tool, however, it would not be necessary for the method to provide results that reliably and quantitatively predict the relative potential for a sampled material to release asbestos under real-world conditions.

Note that, as it specifically pertains to the North Ridge Estates Site, because we are not recommending further application of the Glove Box Method at the site, none of the studies suggested above would further serve efforts to understand exposure and risk at North Ridge. Therefore, it would make sense to fund such research-oriented studies under mechanisms not associated with the North Ridge Estates Site.

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TABLES AND FIGURES

TABLE 1:
COMPARISON BETWEEN PERFORMANCE OF GLOVE BOX AND ELUTRIATOR BASED ON STRUCTURE COUNTS FROM PAIRED ANALYSES

				Glove Box Data					*Elutriator Data*					Comparison Based on Structure Count		
					Number of Structures					Number of Structures						
Elutriator ID Number	**Glove Box ID Number**	**ACM Content (g/g)**	**Asbestos Type**	**Analytical Sensitivity (s/L)**	**Total ISO**	**Total Protocol**	**Long Protocol**	**PCME fibers**	**Analytical Sensitivity (s/g)**	**Total ISO**	**Total Protocol**	**Long Protocol**	**PCME fibers**	**RPD[a]**	**Agreement[b]**	**Relative Sensitivity**
3090503	4114023	0.043	Chrysotile	4.82	132			3	1.97E+06	6				183	Poor	Glove Box more sensitive
3090508	4084103	0.034	Chrysotile	0.91	20				1.38E+06	11				58	Good	Approximately equal sensitivity
3090509	4114030	0.023	Chrysotile	1.27	158			5	1.02E+06	4				190	Poor	Glove Box more sensitive
3090512	4114015	0.014	Chrysotile	4.79	143			3	1.26E+06	9				176	Poor	Glove Box more sensitive
			Amosite							1				NA	Good[c]	Approximately equal sensitivity
3090500	4124112	0.0086	Chrysotile	0.90	1	1		1	1.84E+06	7				150	Poor	Elutriator more sensitive
3090519	4114004	0.0068	Chrysotile	0.89	83			8	1.46E+06	14	1		1	142	Fair	Glove Box more sensitive
3090504	4124106	0.0050	Chrysotile	0.90	136	4	2	5	1.97E+06	28	4	3	2	132	Fair[d]	Glove Box more sensitive
3090514	4080111	0.0012	Chrysotile						1.84E+06	3				NA	Inconsistent[e]	Elutriator more sensitive
			Amosite	0.89	4	3	2	3						NA	Inconsistent[e]	Glove Box more sensitive
3090518	4114011	0	Amosite	0.88	1				1.71E+06	1				0	Good	Approximately equal sensitivity
3090505	4134058	0	Chrysotile	0.76					1.97E+06					NA	Good[c]	Approximately equal sensitivity
3090506	4124119	0	Chrysotile	0.71	2	1		1	1.73E+06	1				67	Good	Approximately equal sensitivity
3090513	4134053	0	Chrysotile	0.72	1				1.44E+06	1				0	Good	Approximately equal sensitivity

Notes:

[a] RPD means Relative Percent Difference, which is defined as: 100*(a-b)/0.5*(a+b). Note that if one of the values are zero, than RPD is undefined.

[b] The indicated agreement is based on the value of the RPD determined for total structures. If RPD < 75, agreement is considered to be "good." If 75 ¨ RPD <150, than agreement is considered "fair." If 150 ¨ RPD, than agreement is considered poor. Given the observed precision of the elutriator method and consideration that two different methods are being compared, these are reasonable precision goals.

[c] In general, when agreement is good, the difference in the observed number of structures can be explained by expected differences due to counting statistics (as described by a Poisson distribution). Even when one of the two values is zero, if the second value is less than 3 (which is the 95% upper confidence limit on a count of zero structures), than agreement can still be considered to be good.

[d] For this one case, the relative counts across other structure size categories do not appear consistent, even if one assumes they remain approximately proportional to total structure counts.

[e] Counts observed across the two methods are clearly inconsistent for this sample. While each detected structures, the type of asbestos detected is inconsistent. Therefore, this cannot be explained by differences in sensitivity.

Working Draft

TABLE 2:
COMPARISON BETWEEN PERFORMANCE OF GLOVE BOX AND ELUTRIATOR BASED ON STRUCTURE CONCENTRATION

Elutriator ID Number	Glove Box ID Number	Asbestos Type	Glove Box Data: Analytical Sensitivity (s/L)	Glove Box Data: Number of Total Structures	Glove Box Data: Concentration (s/L)	Elutriator Data: Analytical Sensitivity (s/g)	Elutriator Data: Number of Total Structures	Elutriator Data: Concentration (s/g)	Concentration Ratio
3090503	4114023	Chrysotile	4.82	132	6.4E+02	1.97E+06	6	1.2E+07	5.4E-05
3090508	4084103	Chrysotile	0.91	20	1.8E+01	1.38E+06	11	1.5E+07	1.2E-06
3090509	4114030	Chrysotile	1.27	158	2.0E+02	1.02E+06	4	4.1E+06	4.9E-05
3090512	4114015	Chrysotile	4.79	143	6.8E+02	1.26E+06	9	1.1E+07	6.0E-05
		Amosite			0		1	1.3E+06	
3090500	4124112	Chrysotile	0.90	1	9.0E-01	1.84E+06	7	1.3E+07	7.0E-08
3090519	4114004	Chrysotile	0.89	83	7.4E+01	1.46E+06	14	2.0E+07	3.6E-06
3090504	4124106	Chrysotile	0.90	136	1.2E+02	1.97E+06	28	5.5E+07	2.2E-06
3090514	4080111	Chrysotile			0	1.84E+06	3	5.5E+06	
		Amosite	0.89	4	3.6E+00			0	
3090518	4114011	Amosite	0.88	1	8.8E-01	1.71E+06	1	1.7E+06	5.1E-07
3090505	4134058	Chrysotile	0.76		0	1.97E+06		0	
3090506	4124119	Chrysotile	0.71	2	1.4E+00	1.73E+06	1	1.7E+06	8.2E-07
3090513	4134053	Chrysotile	0.72	1	7.2E-01	1.44E+06	1	1.4E+06	5.0E-07

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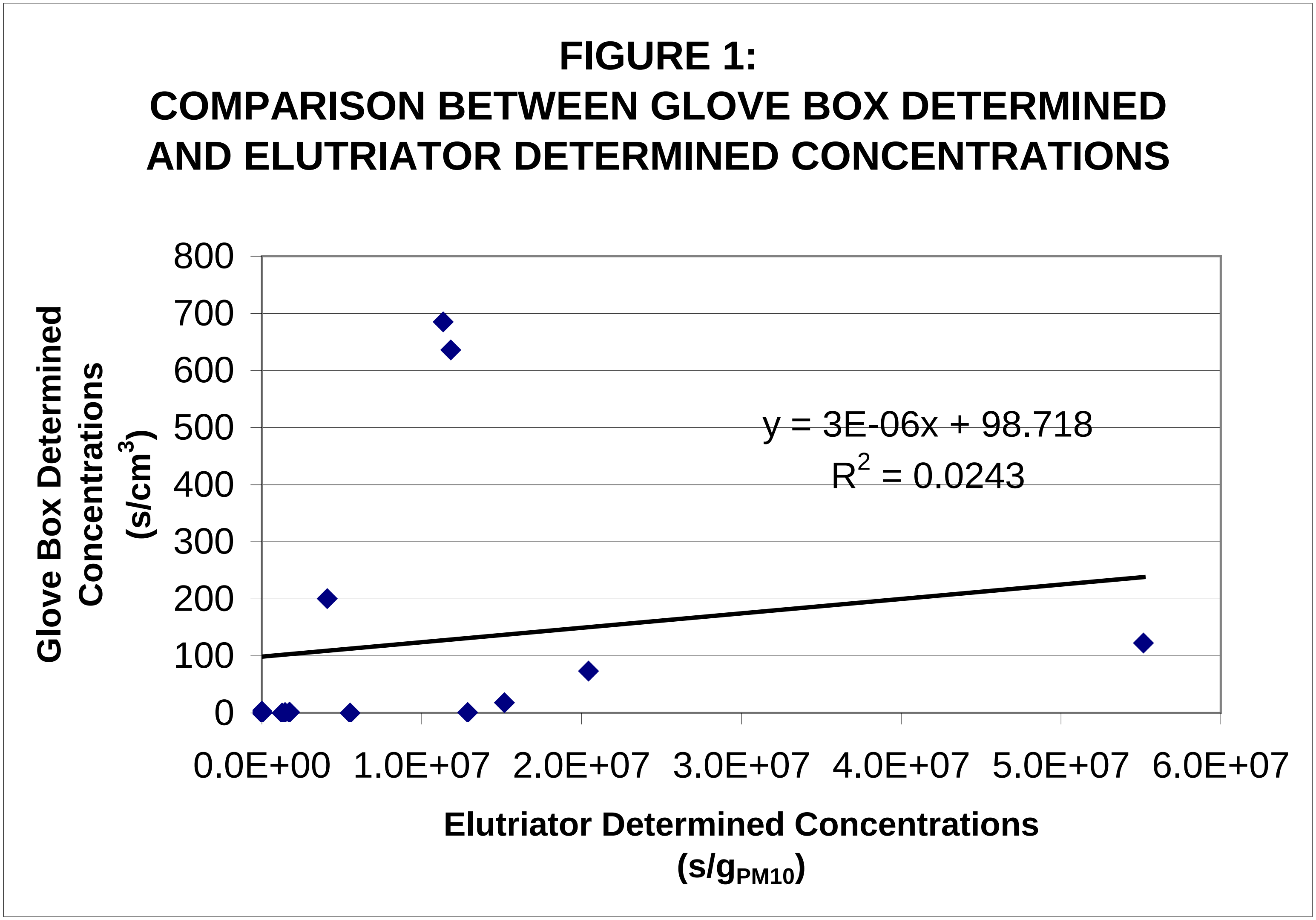
FIGURE 1:
COMPARISON BETWEEN GLOVE BOX DETERMINED
AND ELUTRIATOR DETERMINED CONCENTRATIONS
Glove Box Determined Concentrations (s/cm3)
800
700
600
500
400
300
200
100
0
y = 3E-06x + 98.718
R2 = 0.0243
0.0E+00
1.0E+07
2.0E+07
3.0E+07
4.0E+07
5.0E+07
6.0E+07
Elutriator Determined Concentrations
(s/gPM10)